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1032.057



PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

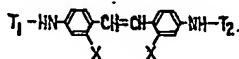
Process for the manufacture of White Pigments

We, CIBA LIMITED, A Swiss Body Corporate, of Basle, Switzerland, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

5 The present invention provides a process for the manufacture of white pigments that contain a brightener and are fast to light which comprises curing the melamine-formaldehyde condensation product in an aqueous mixture containing an optical brightener which is a stilbene derivative comprising at least one acid group imparting solubility in water, a white inorganic pigment derived from beryllium, magnesium, calcium, strontium, barium, zinc, cadmium, mercury, aluminium, gallium, indium or thallium (preferably calcium, strontium, barium, zinc or aluminium) and a colourless water-insoluble, curable melamine-formaldehyde condensation product and comminuting the resulting mass.

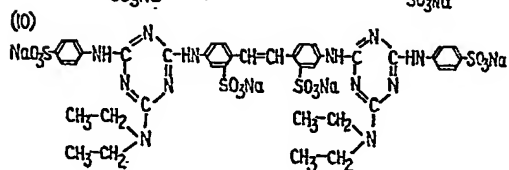
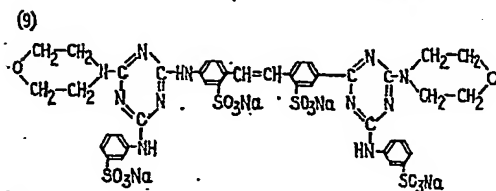
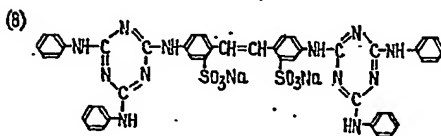
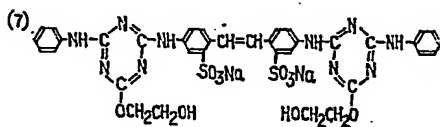
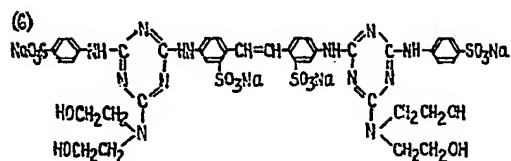
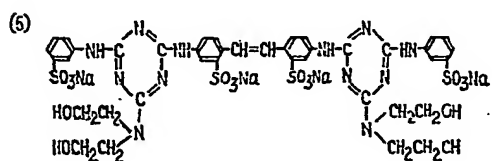
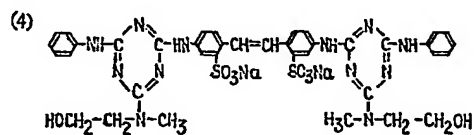
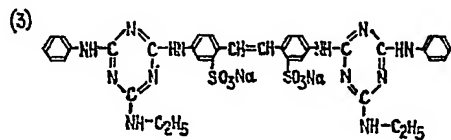
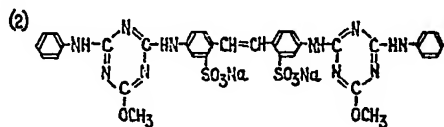
15 The present process is performed with inorganic white pigments of the aforesaid metals. Suitable white pigments of the type defined are, for example, magnesium oxide, calcium oxide, calcium sulphate, strontium oxide, strontium sulphate, zinc oxide and aluminium oxide. Particularly good results have been obtained with the so-called lithopones, that is to say, mixtures of barium sulphate and zinc sulphide, and also with calcium carbonate, zinc sulphide and barium sulphate.

20 The optical brighteners which is a stilbene derivative may be, for example, the triazinylaminostilbene brighteners containing at least one carboxyl group or preferably a sulphonic acid group, and among these the bis-triazinylaminostilbenes of the formula:



(1)

25 where X represents a carboxyl or preferably a sulphonic acid group, and T_1 and T_2 each represents an s-triazin-2-yl nucleus, deserve special mention. Examples of such brighteners, which give excellent results when used in the present process, are the compounds of the formulae:



As melamine-formaldehyde condensation products to be used in the present process there may be specially mentioned those which are obtainable in known manner by condensing melamine with formaldehyde. Particularly suitable condensation products of this type can be prepared, for example, by condensing 1 mol of pure melamine with about 2 to 3 mols of formaldehyde in a weakly basic aqueous medium.

To manufacture the aqueous mixtures the constituents may be mixed in any desired sequence. For example, according to a preferred variant, an aqueous solution of the melamine-formaldehyde condensation product is mixed with the aqueous solution of the stilbene brightener, and the inorganic white pigment is then stirred into the mixture.

The proportions of the ingredients may vary within relatively wide limits. Suitable proportions can be easily established by preliminary experiments. Preferred aqueous mixtures contain for every 100 parts by weight of the inorganic white pigment 0.5 to 5 parts by weight of the afore-mentioned brightener and 50 to 200 parts by weight of the melamine-formaldehyde condensation product.

The melamine-formaldehyde condensation product can be cured even at a low temperature, for example at 20° to 30° C., if desired in the presence of a curing catalyst, in the absence of which a relatively longer curing period is required. Such a curing treatment may produce the cured resin in the form of minute particles so that in most cases further comminution will not be needed. However, it is of advantage to cure the melamine-formaldehyde condensation product by subjecting the aqueous mixture to a treatment at an elevated temperature ranging from 50° to 200° C., preferably from 60° to 120° C., during which water escapes, and the resulting cured mass is subsequently ground to form a fine powder of the desired grain size.

The white pigments obtained by the process of this invention have considerable fastness to light and considerable brilliance. They may be used as white pigments for a wide variety of purposes, for example in making paints (emulsion colours, glue colours, oil colours, varnishes), in the paper industry, in the manufacture of printing inks and leather-covering dyes, and in the manufacture of white-pigmented panels, films, foils, shaped articles or fibres; in the latter application the new white pigments may be added to the moulding, casting or spinning compositions from which the articles are manufactured, or they may be incorporated in previously shaped articles, for example films, foils or ribbons, or they may be used in conjunction with suitable bonding agents for surface treatments. In this manner pure, brilliant white effects are obtained which are fast to light.

Parts and percentages in the following Examples are by weight.

EXAMPLE 1.

100 Parts of a water-soluble melamine-formaldehyde condensation product (obtained by condensing 1 mol of pure melamine with 2 to 3 mols of formaldehyde in a weakly basic aqueous medium and spray-drying the solution) are dissolved in 100 parts of water, and the solution is mixed with 0.7 part of the stilbene brightener of the formula (2) dissolved in a small amount of water. 100 Parts of a lithopone containing 70% of barium sulphate and 30% of zinc sulphide are then stirred in. The resulting viscous mass is then cured for 2 hours at 80° C. and ground to a fine powder.

The white pigment obtained in this manner is outstandingly fast to light. It may be used, for example, for the manufacture of paints.

Similar results are obtained when the stilbene brightener of the formula (2) is replaced by the product of the formula (4), (5), (8) or (10).

EXAMPLE 2.

100 Parts of the melamine-formaldehyde condensation product described in Example 1 are dissolved in 100 parts of water, and a solution of 1.8 parts of the stilbene brightener of the formula (2) in a small amount of water is added. 100 Parts of barium sulphate or calcium carbonate in powder form are then stirred in. A viscous mixture is obtained which is cured for 2 hours at 80° to 90° C., and the hard mass is then ground to a fine powder.

The white pigments obtained in this manner display a pure, brilliant white and outstanding fastness to light. They can be used, for example, for the manufacture of wallpaper printing inks.

Similar results are obtained when in this Example the stilbene brightener of the formula (2) is replaced by the product of the formula (3), (4), (6) or (7).

EXAMPLE 3.

A solution of 100 parts of the melamine-formaldehyde condensation product described in Example 1 in 100 parts of water is mixed with a solution of 3.5 parts of the stilbene brightener of the formula (2), (3), (4), (9) or (10) in a small amount of water. 100 Parts of a zinc sulphide pigment (Registered Trade Mark "Sachtolith") are then stirred into the aqueous mixture. A viscous mass is obtained which is cured for about 2½ hours at 75° to 85° C. The cured mass is then comminuted to a fine powder.

The pigments obtained in this manner have a substantially increased white content and are very fast to light.

When 2 to 5 parts of the above white pigments are incorporated in 100 parts of polyethylene, the latter is rendered a pure, brilliant white of high fastness to light.

WHAT WE CLAIM IS:—

1. A process for the manufacture of white pigments that contain a brightener and are fast to light, which comprises curing the melamine-formaldehyde condensation product in an aqueous mixture containing an optical brightener which is a stilbene derivative comprising at least one acid group imparting solubility in water, a white inorganic pigment derived from beryllium, magnesium, calcium, strontium, barium, zinc, cadmium, mercury, aluminium, gallium, indium, or thallium, and a colourless, water-soluble, curable melamine-formaldehyde condensation product, and comminuting the resulting mass.

2. A process as claimed in claim 1, wherein the melamine-formaldehyde condensation product is cured by heating the aqueous mixture and removing the water.

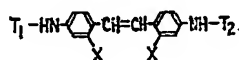
3. A process as claimed in claim 1 or 2, wherein the melamine-formaldehyde condensation product is cured at 60° to 120° C.

4. A process as claimed in one of claims 1 to 3, wherein the melamine-formaldehyde condensation product is one prepared using 1 mol of melamine for each 2 to 3 mols of formaldehyde.

5. A process as claimed in any one of claims 1 to 4, wherein the white inorganic pigment is a lithopone, barium sulphate, zinc sulphide or calcium carbonate.

6. A process as claimed in any one of claims 1 to 5, wherein the optical brightener is a triazinyl-aminostilbene containing at least one acid group imparting solubility in water.

7. A process as claimed in claim 6, wherein the optical brightener is a bis-triazinyl-aminostilbene of the formula:



where X represents a carboxyl or sulphonic acid group, and T₁ and T₂ each represents an s-triazin-2-yl nucleus.

8. A process as claimed in any one of claims 1 to 7, wherein the aqueous mixture contains, for every 100 parts by weight of the white inorganic pigment, 0.5 to 5 parts by weight of the optical brightener, and 50 to 200 parts by weight of the melamine-formaldehyde condensation product.

9. A process for the manufacture of white pigments as claimed in claim 1, substantially as hereinbefore described.

10. A white pigment obtained by the process claimed in any one of claims 1 to 9.

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